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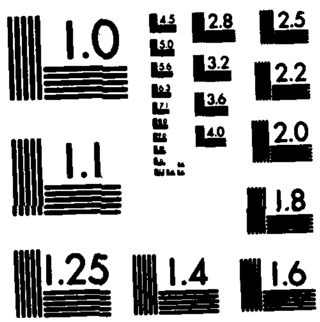


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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Homogeneous crystals of $Cd_{.95}Mn_{.05}Se$ of high optical quality have been grown by a modified Bridgman method. Magnetic susceptibility measurements verify the uniform distribution of Mn(II) obtained after annealing at 600°C. Crystals grown in the presence of 5 atomic percent excess selenium showed high resistivity; the addition of 1 mg iodine to a 10 g charge resulted in n-type conductivity and a room-temperature carrier concentration of $2.9 \times 10^{16} cm^{-3}$. The Hall mobility of these crystals was approximately 290 $cm^2V^{-1}sec^{-1}$.

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Preparation and Characterization of

$Cd_{.95}Mn_{.05}Se$ Single Crystals

by

B. Khazai, R. Kershaw, K. Dwight, and A. Wold

Department of Chemistry

Brown University

Providence, Rhode Island 02912

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# PREPARATION AND CHARACTERIZATION OF $Cd_{.95}Mn_{.05}Se$ SINGLE CRYSTALS

B. Khazai, R. Kershaw, K. Dwight, and A. Wold
Department of Chemistry, Brown University
Providence, Rhode Island 02912

ABSTRACT:

Homogeneous crystals of $Cd_{.95}Mn_{.05}Se$ of high optical quality have been grown by a modified Bridgman method. Magnetic susceptibility measurements verify the uniform distribution of Mn(II) obtained after annealing at 600°C.

Crystals grown in the presence of 5 atomic percent excess selenium showed high resistivity; the addition of 1 mg iodine to a 10 g charge resulted in n-type conductivity and a room-temperature carrier concentration of $2.9 \times 10^{16}$ $cm^{-3}$. The Hall mobility of these crystals was approximately 290 $cm^2 V^{-1} sec^{-1}$.

## Introduction

Recently, cadmium selenide has been the subject of intensive investigation for the characterization of its electro-optical properties(1-5). Joshi et al. (6) have indicated that for cadmium telluride, the characteristic optical response of the photoconductor can be tuned by the incorporation of Mn(II)($3d^5$). Similar phenomena should be observed for cadmium selenide, with the transition energy gap adjusted by the introduction of controlled quantities of manganese dopant.

Crystallographically, one of the modifications of both MnSe and CdSe is that of the wurtzite structure (7), where the cations are tetrahedrally coordinated in a hexagonal close-packed array in which one half of the tetrahedral sites are occupied by the metal atoms. The structural similarity between MnSe and CdSe should therefore allow for the formation of a solid solution of the type $Cd_{1-x}Mn_xSe$. Wiedemeier and Sigai (8) have prepared such a solid solution containing up to 50 atomic percent of manganese and crystallizing with the wurtzite structure.

In this study, we are reporting the growth and characterization of single crystal boules of the general composition $Cd_{1-x}Mn_xSe$. The materials were prepared with either high or low resistivities, and the manganese doping was kept at 5 atomic percent in order to minimize manganese-manganese interactions.

## Experimental

### Crystal Growth

Single crystals of the system $Cd_{1-x}Mn_xSe$ were prepared from the melt, using the Bridgman technique. The starting materials were in their elemental state and were subjected to purification prior to their use. Cadmium (Johnson Matthey 99.999%) was heated to the melting point under a dynamic vacuum for a few minutes. Selenium was purified by sublimation of the element (Atomergic Chemetals 99.9999%) at 200°C and under a dynamic vacuum. Manganese (Atomergic Chemetals 99.99%) was deoxidized by placing the metal in a sealed silica tube along with titanium metal in a separate compartment. The tube was then heated at 1000°C for 24 hours.

The charge containing approximately 10 g of stoichiometric amounts of the starting materials was placed in a heavy-wall silica tube and sealed under high vacuum. The tube was then placed in a vertical furnace equipped with a puller/rotor action motor. The rotor was used to ensure uniform heating across the growth ampoule during crystal formation. The charge was allowed to prereact overnight in the hot zone of the furnace at 1000°C before increasing the temperature to 1200°C. The growth ampoule was subsequently raised above the hot zone and was allowed to pass through a temperature gradient at a rate of 3.2 cm/day for a period of five days. The best crystals were obtained when 5 atomic percent excess selenium was used. The crystal grew approximately along the c axis. The angle between the c axis and the growth direction is $\sim 15°(\pm 1°)$. The lattice parameters are $a = 4.268(2)$Å, $c = 6.983(2)$Å, and the space group is $P6_3mc$.

Conducting crystals were prepared by the introduction of small quantities of iodine into the charge either through an H-tube, for larger quantities, or by prereacting the selenium with iodine at 100°C in a sealed tube for smaller quantities.

The as-grown crystals possessed a relatively high range of inhomogeneity with respect to manganese distribution from top to bottom of the boules. An equilibrium state could, however, be attained by a subsequent five-day annealing of the growth tube in the constant temperature zone of a wound-core transport furnace at 600°C or 800°C for high resistivity or high conductivity samples, respectively. The crystals thus formed were cut into discs and subjected to various measurements.

### X-ray Analysis

Powdered samples were analyzed on a Philips-Norelco powder diffractometer using $CuK\alpha_1$ ($\lambda = 1.5404$Å) radiation, to ascertain the formation of the cadmium selenide phase.

## Magnetic Measurements

Magnetic susceptibilities were measured using a Faraday balance (9) at a field strength of 10.4 kOe. The data were then corrected for the core diamagnetism of cadmium selenide.

## Electrical Measurements

The electrical measurements were made using the van der Pauw technique (10). Contacts were made by the ultrasonic soldering of indium directly onto the samples, and their ohmic behaviors were established by measuring their current-voltage characteristics. The sign of the majority carriers was determined from the qualitative measurement of the Seebeck effect, as well as from Hall measurements.

## Optical Measurements

Optical transmissions were measured using a tungsten iodide lamp and a calibrated silicon diode. Spectral transmission data were obtained using a monochromator (Oriel Model 7240). No correction was made for the surface reflectivity of the polished specimen.

## Results and Discussion

Crystals of the composition $Cd_{.95}Mn_{.05}Se$ were grown from the melt using the Bridgman technique. High quality, single crystal boules were obtained when 5 atomic percent excess of selenium was introduced into the growth ampoule along with the charge. X-ray diffraction patterns of the samples indicated the formation of single-phase products which could be indexed on the basis of a hexagonal unit cell (11).

Magnetic measurements on sections cut along the growth axis of the crystal indicated regions of inhomogeneity with respect to manganese distribution, extending from top to bottom of the crystal. Such non-uniformities were indicated by differences of as much as $0.6\mu_B$/Mn(II) between top and bottom sections of the crystal boule. However, annealing of these crystals in the growth tube at 600°C resulted in the redistribution of manganese throughout the boule. The measured effective moments corresponded closely to the theoretical value of $5.9\mu_B$, as expected for a localized spin-only moment $d^5$ system (Table I).

Figure 1 indicates the magnetic susceptibility behavior over the range from liquid nitrogen to room temperature. The material shows Curie-Weiss behavior in this region with a small antiferromagnetism as indicated by a Weiss constant, $\theta$, of -28 K.

The electrical measurements of the as-grown or annealed crystals indicated resistivities greater than $10^6\Omega$-cm. Burmeister et al. (3) and Hung et al. (12) have shown that the resistivity of cadmium selenide is related to cadmium or selenium vapor pressures above the specimen; the resistivity increases with increased selenium pressure and decreases with cadmium

TABLE I

Electrical and Magnetic Data on $Cd_{.95}Mn_{.05}Se$

| Crystal | Section | $I_2$(mg) | $\rho_{300K}$($\Omega$-cm) | $\chi_g$($10^{-6}$emu/g) [a] | $\mu_{eff}$ |
|---|---|---|---|---|---|
| I | top | 0 | $>10^6$ | 3.46 | 5.79 |
| | bottom | 0 | $>10^6$ | 3.46 | 5.79 |
| II | top | 1 | 1.94 | 3.58 | 5.88 |
| | bottom | 1 | 0.83 | 3.46 | 5.79 |

a = Corrected for core diamagnetism of CdSe = $0.31 \times 10^{-6}$emu/g.

vapor. This is consistent with the high resistivities observed for the manganese-doped samples grown in the presence of excess selenium. However, annealing under cadmium pressure at 400°C resulted in the formation of non-homogeneous, surface-conducting materials.

Optical transmission data for this system is indicated in Figure 2. Very high transparency is observed in the longer wavelengths as the energy of the incident photons becomes small compared to the optical transition gap which is 1.74 eV for cadmium selenide.

FIG. 1

Temperature dependence of the inverse magnetic susceptibility of $Cd_{.95}Mn_{.05}Se$.

FIG. 2

Variation of optical transmission with wavelength for $Cd_{.95}Mn_{.05}Se$ slices with both high and low resistivity and thicknesses of 0.81 and 0.74 mm, respectively.

FIG. 3

Temperature dependence of the resistivity of conducting $Cd_{.95}Mn_{.05}Se$.

FIG. 4

Variation of Hall carrier concentration with temperature for conducting $Cd_{.95}Mn_{.05}Se$.

FIG. 5

Variation of Hall mobility with temperature for conducting $Cd_{.95}Mn_{.05}Se$.

The crystals prepared from charges containing 1 mg iodine for a 10 g charge showed n-type conductivity, with room temperature resistivities less than 2.0 Ω-cm. Figure 3 indicates the variation of resistivity with temperatures; the resistivity decreases up to about 200°K where it stabilizes and remains essentially constant to room temperature. This behavior may be explained by the exhaustion of the majority charge carriers in the donor level, as well as by losses in mobility due to increased lattice scattering. Analysis of Hall measurement data (Figure 4) indicates that this donor state is very shallow, located 0.03 eV below the conduction band with a carrier concentration of $2.9 \times 10^{16}$ $cm^{-3}$. The Hall mobility of the electrons at room temperature is approximately 290 $cm^2V^{-1}sec^{-1}$ and shows some temperature dependence (Figure 5). This value is approximately half that reported for cadmium selenide crystals grown in the presence of excess cadmium vapor (3) or with an argon atmosphere (13).

The optical transmission data for conducting crystals are included in Figure 2. The behavior is similar to that observed for high resistivity samples; slightly lower transparency was observed at wavelengths approaching the optical energy gap of cadmium selenide. This behavior can be attributed to the presence of low energy transitions to shallow donor levels.

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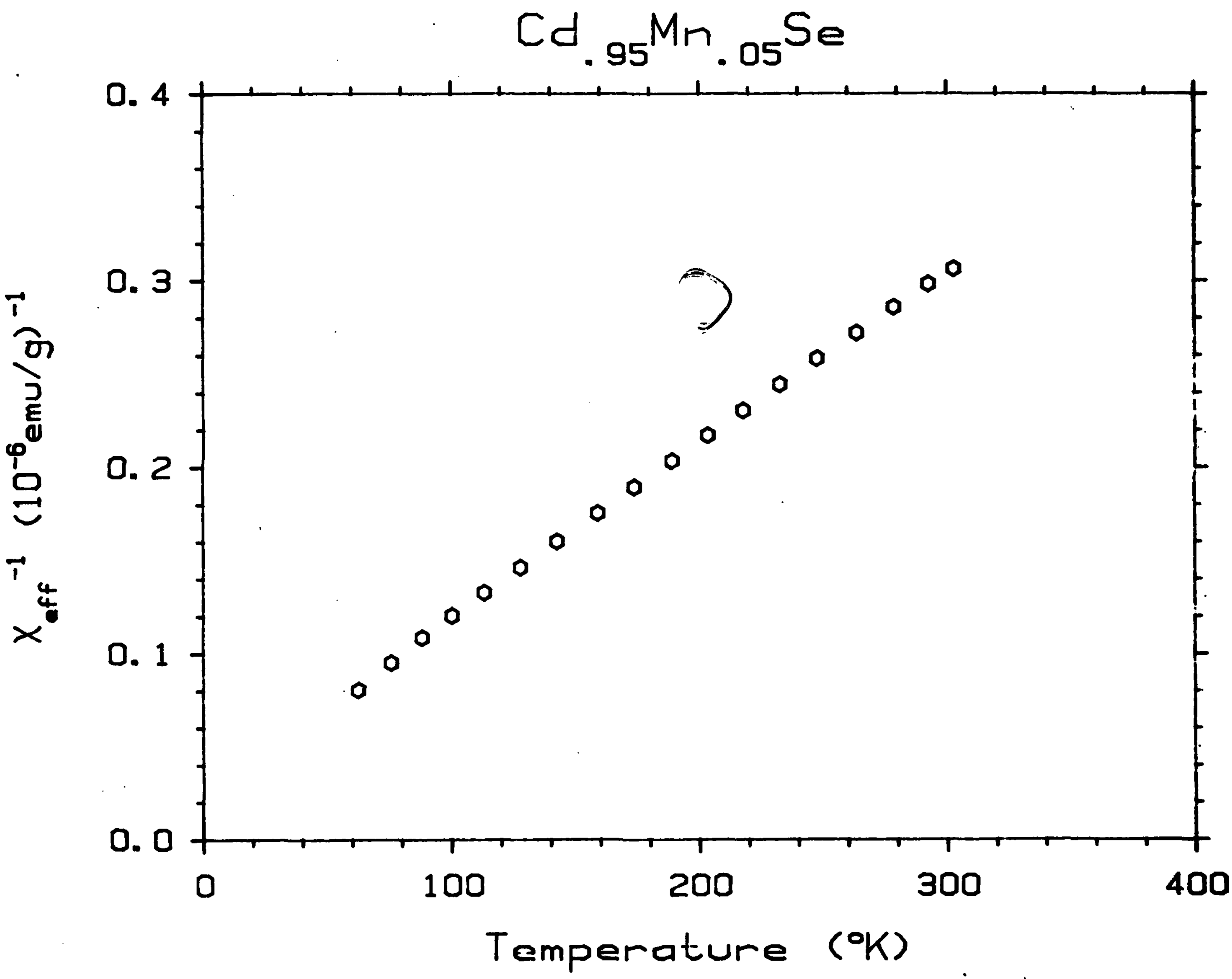
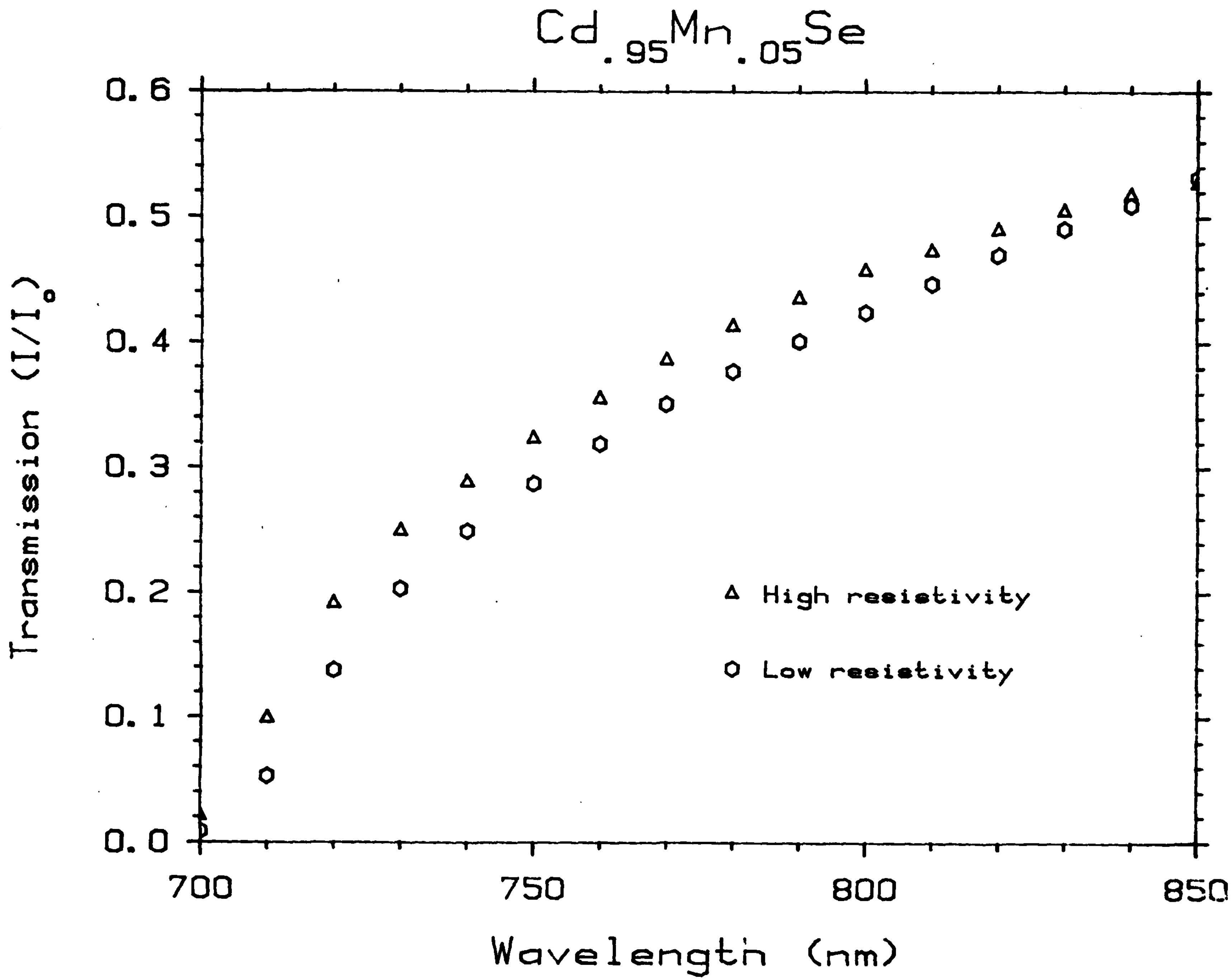
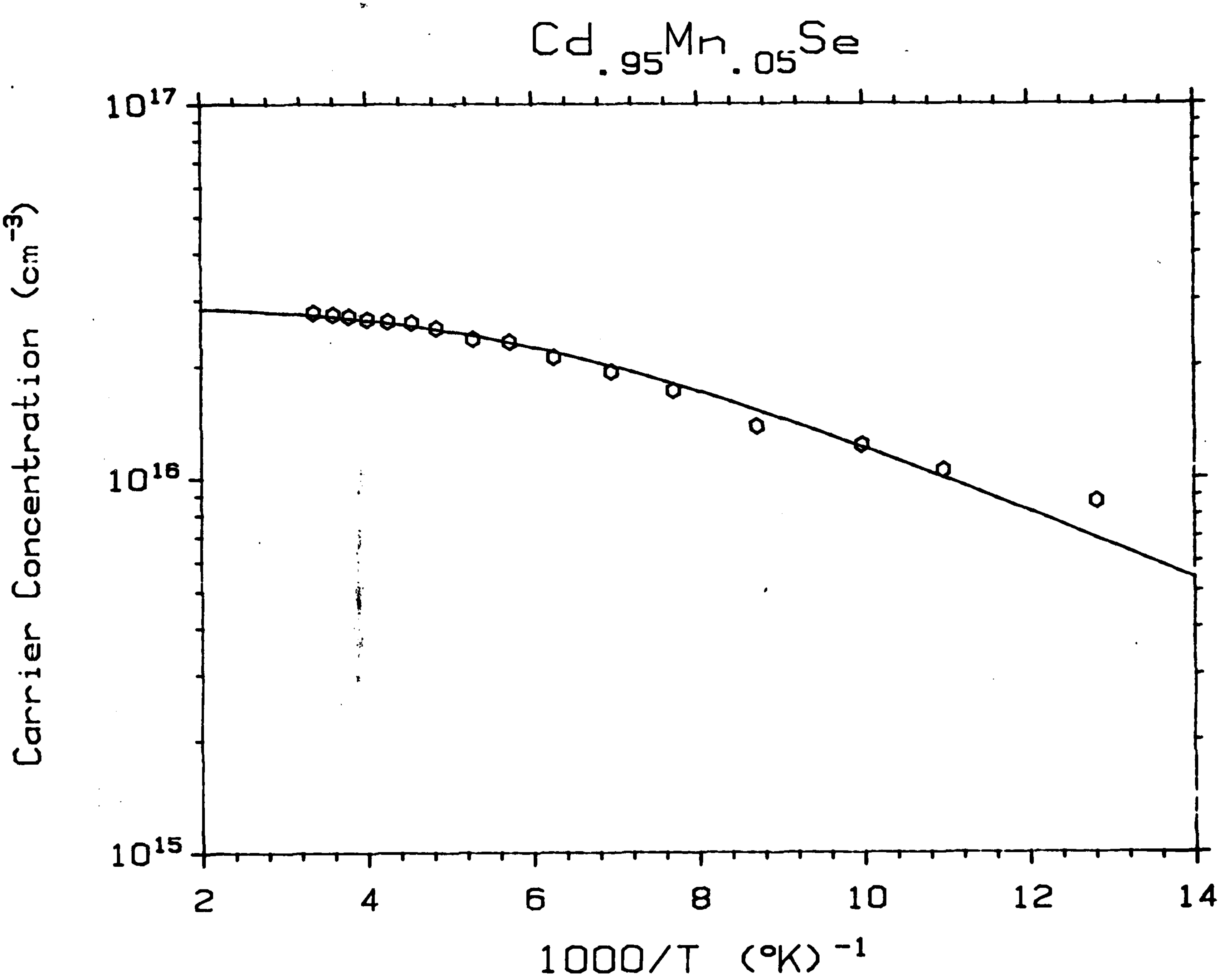
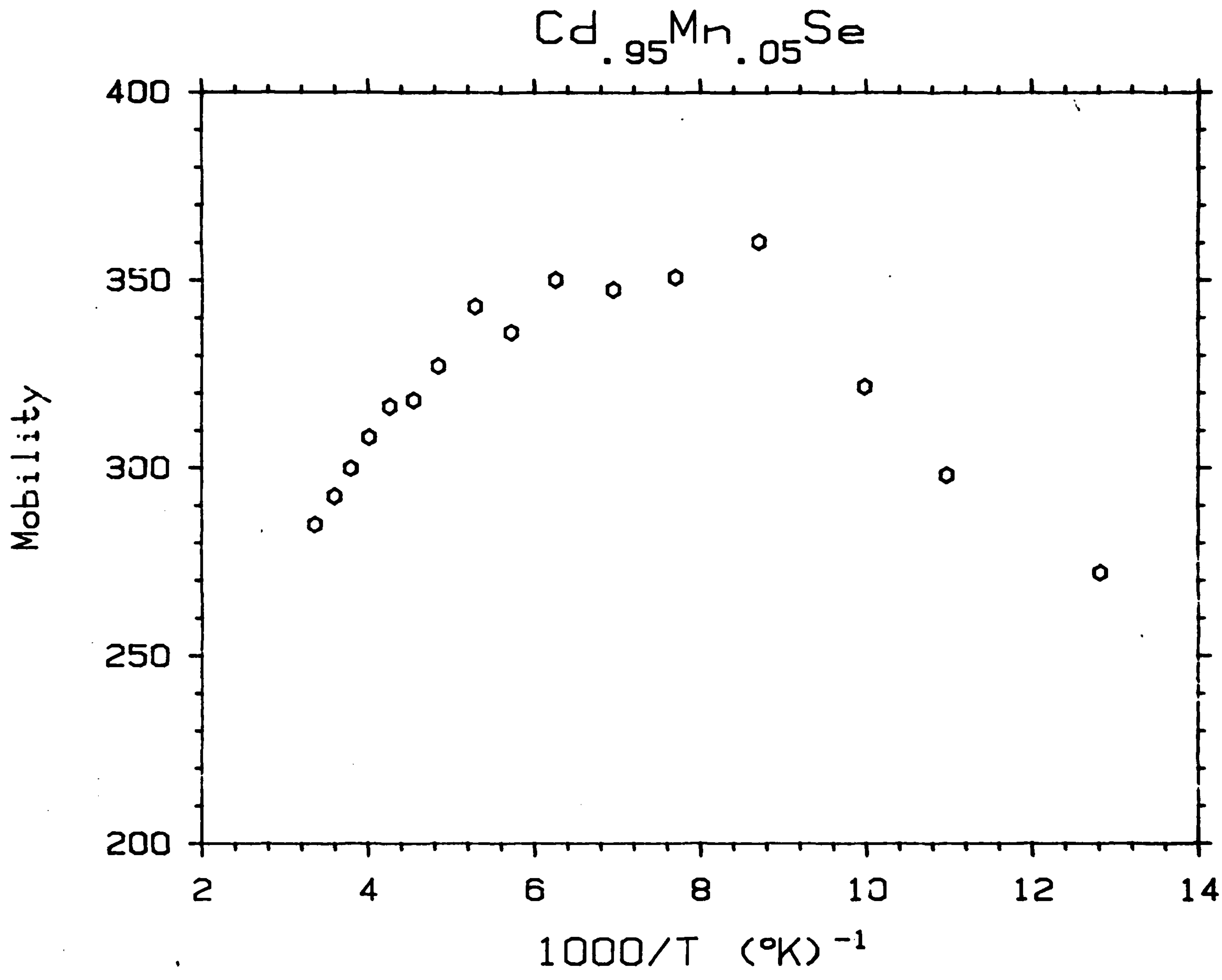

Cd.95Mn.05Se
χeff-1 (10-6emu/g)-1
0.0
0.1
0.2
0.3
0.4
0
100
200
300
400
Temperature (°K)



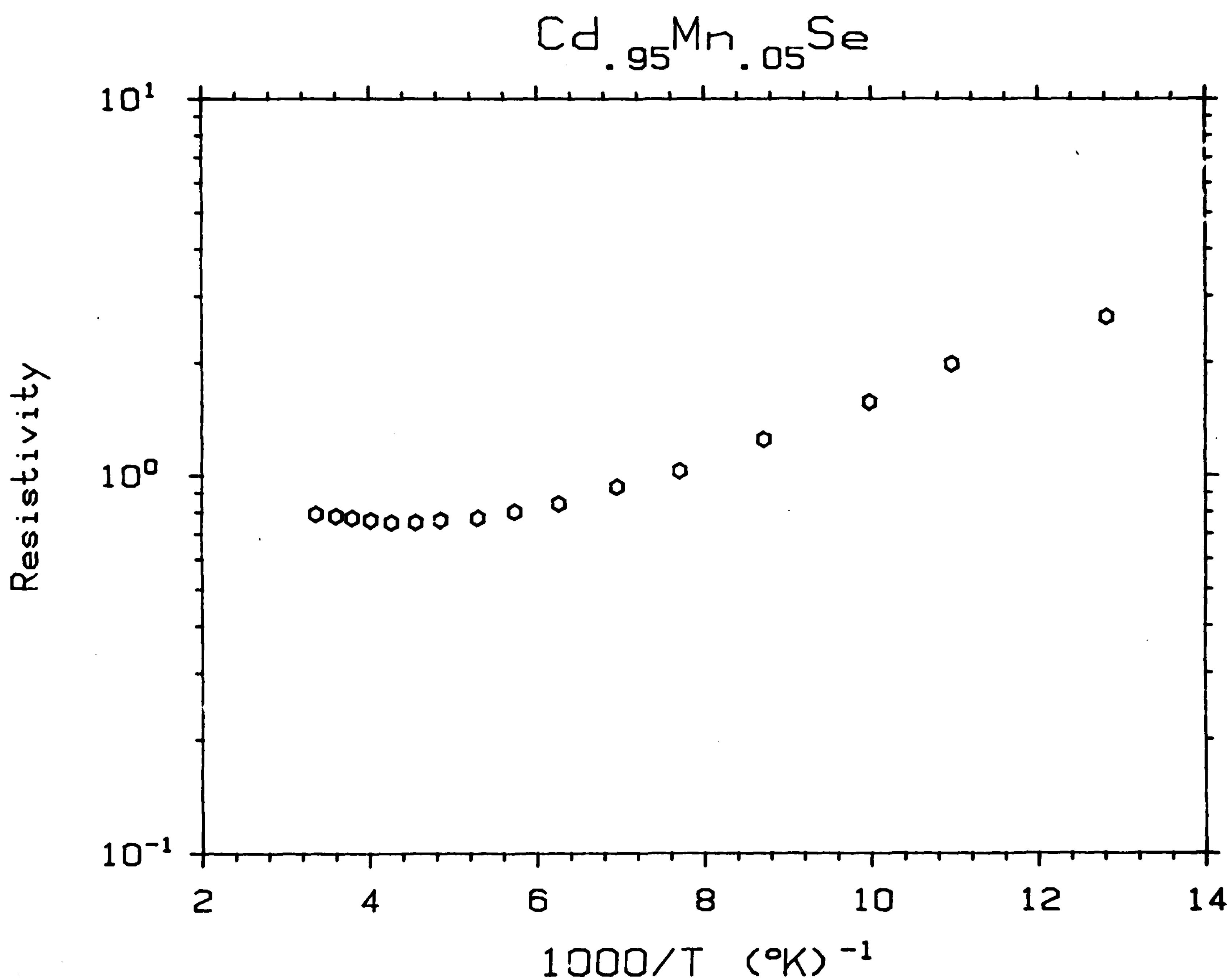
Cd.95Mn.05Se
Transmission (I/Io)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
700
750
800
850
Wavelength (nm)
High resistivity
Low resistivity



Cd.95Mn.05Se
Carrier Concentration (cm-3)
10^17
10^16
10^15
2
4
6
8
10
12
14
1000/T (°K)-1



Cd.95Mn.05Se
Mobility
400
350
300
250
200
2
4
6
8
10
12
14
1000/T (°K)-1



Cd.95Mn.05Se
Resistivity
10^1
10^0
10^-1
2
4
6
8
10
12
14
1000/T (°K)^-1

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